NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

Technical Report 32-1310

Studies on Ethylene Oxide–Freon 12 Decontamination and Dry-Heat Sterilization Cycles

S. H. Kalfayan
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Preface

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Abstract

Several problems related to the ethylene oxide–Freon 12 decontamination process were investigated, including: (1) the quantitative estimation of the ethylene oxide concentration in the decontamination chamber, (2) the possibility of reaction between the gases present in the decontamination chamber, (3) the establishment of adequate air-flushing periods to dissipate the absorbed sterilant gases from the materials, and (4) the determination of moisture content. Results show that (1) gas chromatography can be employed as an analytical method for the quantitative estimation of ethylene oxide, with fairly good accuracy, (2) ethylene glycol and small amounts of HCl are formed in the decontamination chamber, (3) air-flushing periods should be extended to 8 h or more for the effective dissipation of absorbed sterilant gases, and (4) none of the commercial moisture-sensing instruments are satisfactory for determining the relative humidity in an atmosphere of ethylene oxide–Freon 12. The effect of various dry-heat sterilization cycles on a selected number of polymeric materials was also investigated, and the results were compared with those obtained for dry-heat sterilization set forth in the JPL specification. Indications were that polymeric materials exposed to higher temperatures for shorter periods—i.e., 155°C for 240 h—were more affected than by exposure to lower temperatures for longer periods—i.e., 105°C for 3600 h. Polymeric materials of known heat resistance were not so affected. Also, the presence of small quantities of oxygen, i.e., 0.45%, in the dry-heat sterilization chamber did not significantly affect the property changes of the exposed polymeric materials.

Studies on Ethylene Oxide–Freon 12 Decontamination and Dry-Heat Sterilization Cycles

I. Introduction

During the past several years, extensive studies have been made on the effects of decontamination and dry-heat sterilization on the properties of hundreds of polymeric products for use on sterilized spacecraft (Refs. 1–5).

The decontamination and dry-heat sterilization procedures used for these studies have been in accordance with three specifications developed at the Jet Propulsion Laboratory. The first of these specifications calls for two preliminary decontamination cycles of 24 h each with ethylene oxide (ETO)–Freon 12 mixture. This is followed by three 36-h cycles of dry-heat sterilization at 145°C in nitrogen, required by the second specification. The third, more recent, specification calls for six 28-h cycles of decontamination with ETO–Freon 12 at 50°C, followed by six 92-h cycles of dry-heat sterilization at 135°C in dry nitrogen. These conditions apply only to the more rigid, *type approval* sterilization testing of components and materials; requirements are less rigorous for the *flight approval* testing.

A. Scope of Work

This report concerns an examination of some of the practices and specified procedures for decontamination, and an investigation of the effects of various time–temperature environmental regimens on polymeric products.

Areas studied concerning the decontamination process were: (1) the estimation of ETO concentration in the decontamination chamber, (2) the investigation of possible reactions between gases present in the decontamination chamber, (3) establishment of optimum air-flushing periods to dissipate the absorbed sterilant gas mixture from materials, and (4) the determination of moisture content.

Investigations concerning the dry-heat sterilization step included the exposure of some selected polymeric products to various sterilization cycles. Although the latest specifications and developmental work on the sterilizability of components and materials have centered around 135°C for six 92-h cycles, other time–temperature regimens would be as effective or acceptable. Two such regimens selected, both of which used nitrogen, were: six 600-h cycles, each at 105°C, and six 40-h cycles, each at 155°C. The effects of these regimens on the properties of the polymeric products were investigated, and the results were compared with those obtained for the 135°C exposure.

In the actual dry-heat sterilization of a landing capsule, it will be difficult and expensive to create an absolutely air-free atmosphere in the thermal sterilization chamber. Since an estimated 0.25 to 0.50% oxygen could be present in the nitrogen-filled oven, it was considered important to find out whether this concentration of oxygen would have any pronounced effects on the properties of polymeric products as compared with the property changes occurring in pure nitrogen at the same temperatures. The results of this study are also discussed in this document.

B. Plan of Report

The description of the investigation and findings are contained in the body of this report. Methods and procedures used in ETO–Freon 12 decontamination are evaluated and discussed in Section II. The effects of various sterilization cycles on selected polymeric products are discussed in Section III.

The various criteria for rating the sterilizability of polymeric materials are presented in Appendix A. The detailed test results for products that were exposed to ETO and various dry-heat sterilization cycles are contained in Appendix B.

II. Ethylene Oxide–Freon 12 Decontamination: Evaluation of Methods and Procedures

A. Quantitative Estimation of the Chamber Gases by Gas Chromatography

The latest JPL specification for decontamination requires a concentration of 600 ±50 mg/l of ETO in the chamber. For a sterilant gas mixture of known composition, the chamber pressure necessary to obtain the desired ETO concentration at a given temperature can be calculated by use of the ideal gas relationships—the method commonly used to establish ETO concentrations (Ref. 6). The drawbacks of the method are in two basic assumptions: it assumes ideal behavior on the part of the sterilant gas mixture [in this case, ETO and dichlorodifluoromethane (Freon 12)] and, also, that there is a known composition of the gas mixture that is introduced into the chamber and remains constant during a run.

Employment of a method independent of such assumptions was desirable for the analysis, so the technique of gas chromatography (GC) was selected to make the quantitative measurement. In addition to the ETO and Freon 12, the decontamination chamber contained moisture, which was necessary for effective decontamination, and a very low concentration of air, which resulted from leakage. The quantitative estimation of moisture by GC could not be achieved; trials with several different columns and analytical conditions could not eliminate the tailing of the water peak. However, the determination of moisture content, or the relative humidity, can be achieved by moisture detectors; the method and apparatus are described under D of this section.

1. Experimental method.

a. Apparatus. An F&M Model 720 chromatograph equipped with a thermal conductivity detector was used in the investigation. Peak areas were obtained with an electronic digital integrator.[1] Samples were introduced into the chromatograph by a model GV-10 gas sampling valve.[2]

[1]Manufactured by Infotronics, Houston, Texas.

[2]Manufactured by F&M Scientific Corp., Avondale, Pennsylvania.

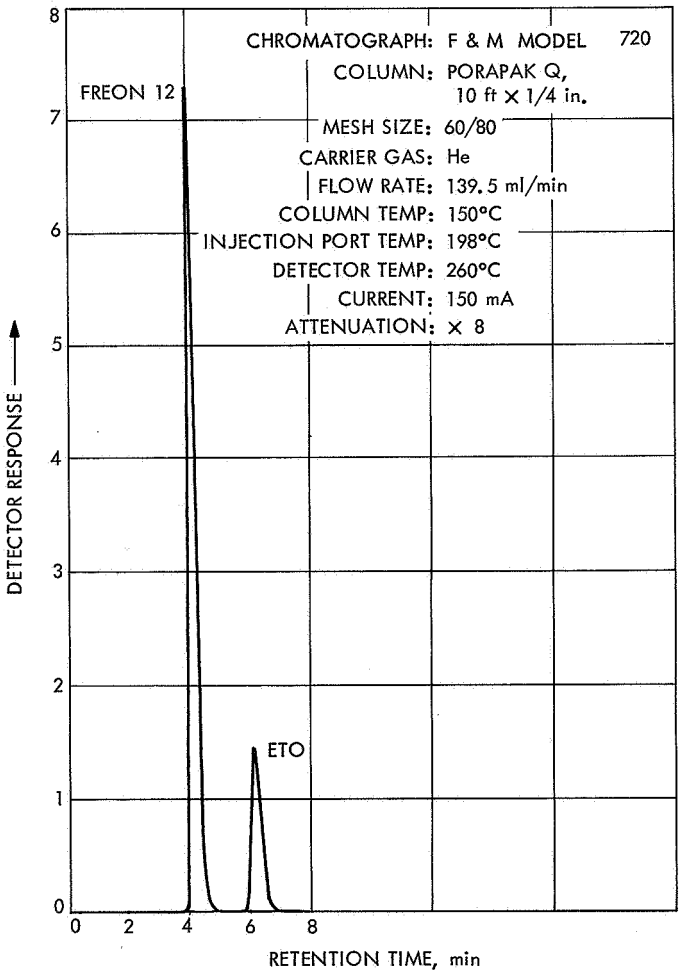

Fig. 1. Chromatogram of sterilant gases

b. Sampling. For both calibration and analysis, the gases were first introduced into 300-ml glass sampling bottles as a convenience in handling. Sampling was made from these bottles. Before injection into the chromatograph, the temperature and pressure of the gas were brought to room conditions.

The column used and the analytical conditions are shown in Fig. 1. The calibration curves shown in Fig. 2, were obtained by use of stainless steel loops of known volumes—which were determined by first filling them with mercury, then weighing the amount of mercury used. The volumes stated in the discussion include the volume of the loops plus the internal volume of the valve, which was 0.27 cc (amount supplied by the manufacturer).

2. *Results and discussion.* The peak area measurements for ETO, either during calibration or an actual analysis, showed standard deviations up to 1.4% of the mean value. For Freon 12, the standard deviations were within 1% of the mean value.

The partial volume of ETO at room conditions, in a known volume of sterilant gas mixture sampled from the chamber, was obtained from its peak area and the calibration curve of Fig. 2. If the density of ETO at chamber conditions of 50°C temperature and 20.7 lb/in.^{2} pressure were known, then its mass at these conditions—and, therefore, its concentration, in milligrams per liter—could be calculated. This computation was possible by utilizing the experimental volumetric data of Walters and Smith (Ref. 7) for superheated ETO; part of the data are shown in Table 1. When the logs of pressures in Table 1 were plotted against the logs of the corresponding densities, a linear relationship was obtained (Fig. 3). The density of ETO at the chamber pressure (20.70 lb/in.^{2} at 50°C) could be calculated from the slope of the curve.

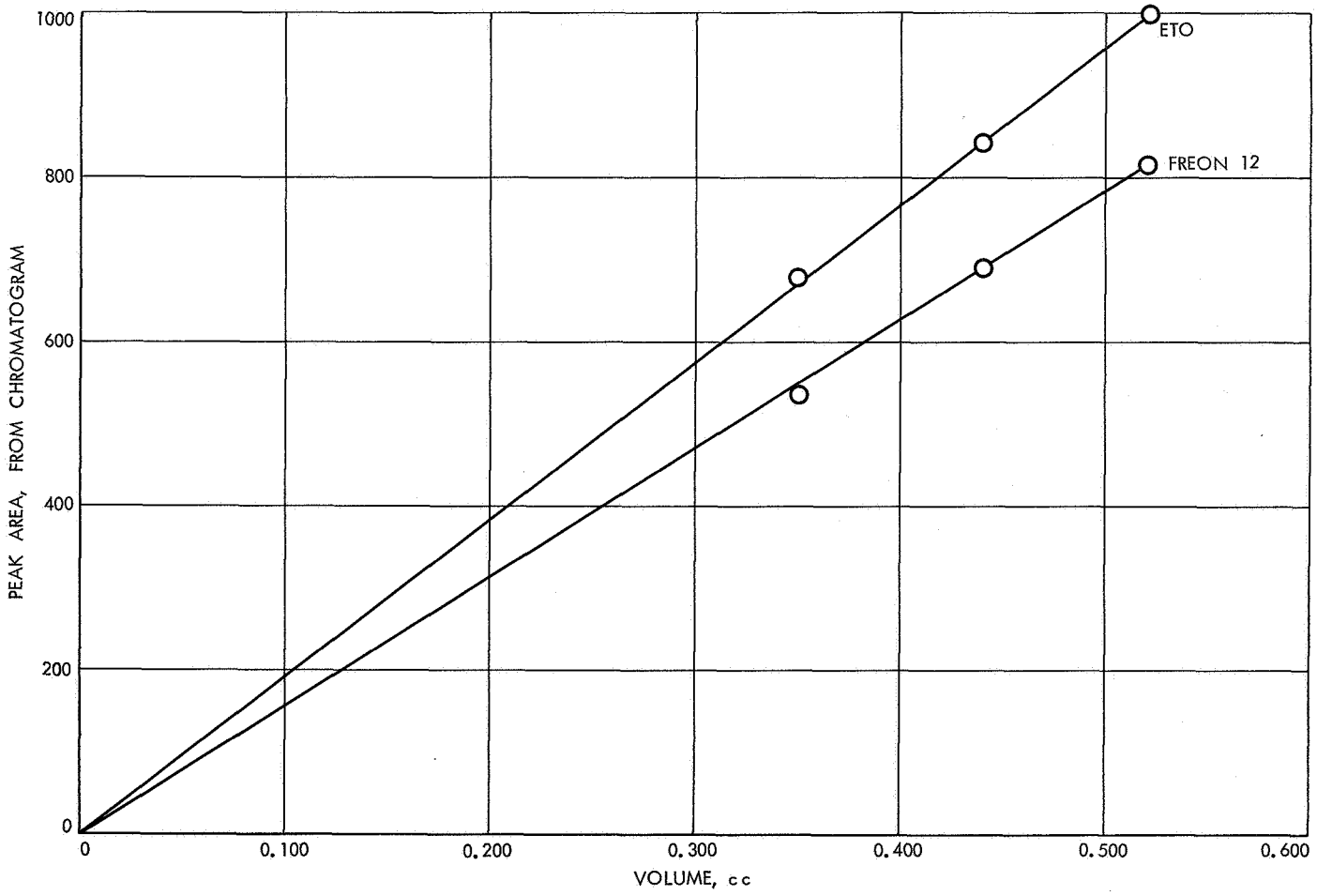

Fig. 2. Calibration curves

Table 1. Properties of ethylene oxide at 50°C

Absolute pressure, lb/in.^{2}	Specific volume,[a] ft^3/lb	Density, lb/ft^3	Density, gm/cc
1.0	141.5	0.007	1.121×10^{-4}
5.0	27.77	0.036	5.766×10^{-4}
14.7	9.423	0.106	1.698×10^{-3}
20.0	6.889	0.145	2.322×10^{-3}
[b]20.7	—	—	[c]2.400×10^{-3}

[a]Obtained from the experimental volumetric data of Ref. 7.
[b]Chamber pressure.
[c]Obtained from plot of Fig. 3.

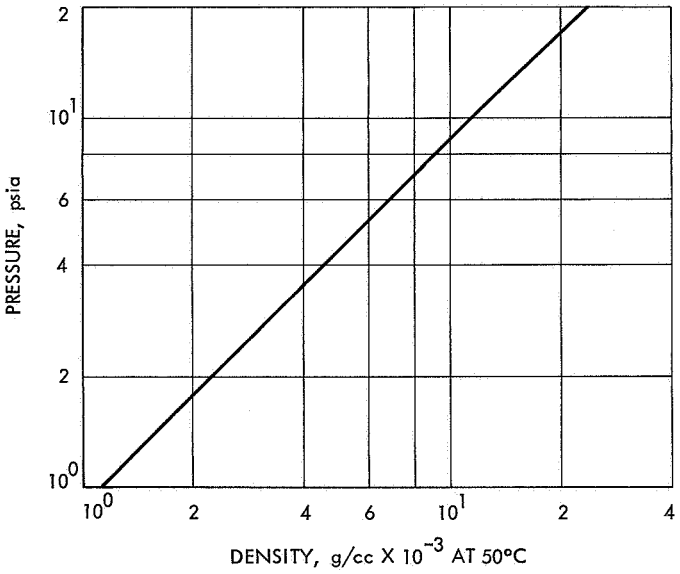

Fig. 3. Relationship of density to pressure of ETO

a. Example of analysis. One actual analysis and calculation made is recorded below. Basic values used were:

Volume of chamber sample injected	0.520 cc
Peak area of ETO	230 ±1.4%
Partial volume of ETO (from calibration curves)	0.122 cc ±1.4%
Density of ETO at 50°C and 20.70 lb/in.^{2}	2.40×10^{-3} g/cc or 2.40 mg/cc

Into the equation

$$\frac{\text{partial volume of ETO (cc)} \times \text{density (mg/cc)}}{\text{volume of injected sample (cc)}} \times 1000 = \text{mg/l ETO}$$

substitution of quantities gives

$$\frac{0.122 \times 2.40}{0.520} \times 1000 = 563 \pm 8 \text{ mg/l ETO}$$

From the ideal gas relationship,

$$w = \frac{PVM}{RT} \tag{1}$$

where

w = weight of ETO in sample analyzed, mg

P = chamber pressure, atm

M = molecular weight of ETO, mg

R = gas constant, l-atm/°K

T = temperature, °K

to compute the concentration of ETO at chamber conditions, the value 549 ±8 mg/l was obtained. This value is 2.3% lower than that obtained using the experimental volumetric data; such variation can be expected for an easily condensible gas such as ETO.

The concentration in mg/l is obtained from Eq. (1) thus,

$$\text{concentration (mg/l)} = \frac{w}{0.520 \text{ cc sample}} \times 10^3$$

The concentration of ETO obtained was lower than expected for a mixture supposed to have an original 27.3% by volume ETO concentration. Analysis of the sterilant gas mixture in the expansion tank, where the partially liquid sterilant mixture is expanded into gaseous form prior to entrance into the decontamination chamber, showed a concentration of 23% ETO by volume.

When a sample taken from the supply cylinder itself was analyzed, the ETO concentration was found to be 29% by volume.

b. Difficulty in verifying mixture composition. These findings indicate a problem in tapping the sterilant mixture from its container. The rate at which the liquid mixture is vaporized, fractionation of the gaseous components and, possibly, some polymerization of the ETO, tend to alter the composition of the sterilant gas. It cannot be shown from these results that the manufacturer's stated composition of the mixture, i.e., 27.3% ETO and 72.7% Freon 12 by volume, is in error.

3. Conclusions. Results obtained from this study allow the following conclusions: (1) It is not possible to rely on assumptions made as to the composition of the sterilant gas mixture introduced into the decontamination chamber, although the composition of the liquid in the cylinder may be known accurately. (2) Actual analysis of the ETO in the chamber is necessary to establish the required concentration. (3) Gas chromatography can be employed as an analytical method, with fairly good accuracy.

B. Reactions in the Decontamination Chamber

The ETO decontamination procedure according to the JPL specification is carried out at 50°C and 50% relative humidity for six cycles of 28 h each. It was suspected that, under these conditions, ETO might react with moisture to form ethylene glycol, and also, that Freon 12 (dichlorodifluoromethane) could hydrolyze to form HCl, especially in the presence of acidic or basic impurities originating from the polymeric materials.

1. Experimental method. To test this possibility, five 250-ml-capacity ampules were filled with the chamber gases. Three of the ampules, containing only the chamber gases, a brass strip, and strips of polymeric materials, respectively, were subjected to 50°C for 180 h. The other two ampules, one containing a brass strip and the other strips of polymeric materials, were subjected to 58°C for 180 h.

2. Results and discussion. After the heating period, analysis by mass spectrography showed a peak at mass 36 in all five ampules. The mass-36 peak, considered to be HCl, amounted to 0.06 mole %, in the case of ampules subjected to 50°C; the amounts were 0.08 and 0.14 mole %, in the cases of ampules subjected to 58°C. The higher mole percent was obtained from the ampule containing the brass strip.

Only a trace of mass-36 peak was shown with the ETO–Freon 12 mixture sampled directly from the original gas cylinder.

It was concluded that HCl was formed in small amounts under the conditions of the decontamination process. This experiment indicated that the presence of polymeric products did not influence the formation of HCl; but the presence of brass and increased temperature increased the rate of HCl formation.

Infrared spectrographic analysis of droplets obtained from one of the ampules showed the presence of ethylene glycol.

C. Establishing Optimum Air-Flushing Periods

At the end of each ETO–Freon 12 exposure cycle, the chamber is evacuated and flushed with ambient air for 2½ to 2¾ h to free it and the decontaminated materials from sterilant gases.

There was strong indication from experience with the ETO decontamination of polymeric products that flushing for 2½ to 3 h with air still left considerable amounts of absorbed sterilant gas mixture in the polymer samples. It was necessary to establish a more effective flushing period since, shortly after the ETO decontamination, samples were exposed to dry-heat sterilization. The absorbed gases could damage the polymeric products at the higher temperature, before thay had a chance to be desorbed.

1. Experimental method. The experimental approach took the following form: five uniform-size strips were cut from each of five representative polymeric products (see Figs. 1 and 2) and weighed. They were then exposed to one cycle of ETO–Freon 12 decontamination, per specifications. Sample strips from each product were taken out of the chamber before any flushing and were placed in separate ampules and sealed. This process was repeated after periods of 2, 4, 6, and 8 h of flushing. The weight of the unflushed samples, and those flushed for 8 h were measured. The ampules were left for 24 h to provide enough time for desorption before the gases were analyzed by gas chromatography. Only Freon 12 and ETO were considered.

2. Results and discussion. The results of this experiment are plotted in Figs. 4 and 5. The values of the peak areas for Freon 12 (Fig. 4) and ETO (Fig. 5) obtained for unflushed samples (zero hour) were set to equal 100%. Thus, the peak area obtained for Freon 12 from the analysis of the ampule containing the Stycast epoxy product (Fig. 4) after 2 h of flushing, was 80% of the area obtained from the ampule containing the unflushed sample. Failure to detect any Freon 12 or ETO in any of the ampules did not necessarily mean that the sample strip was freed of these gases after a period of flushing. With the exception of the glass-filled phenolic compound, all other four compounds still showed more weight after 8 h of flushing than they did before exposure to the decontamination process, although no gases could be detected after this period for three of the compounds—namely, the RTV silicone, the Stycast epoxy, and Tedlar. Weight measurements showed that these three compounds still retained, after 8 h of flushing, 5 to 10% of the total weight gained. The polyurethane product retained about 40% of its gained weight after 8 h of flushing.

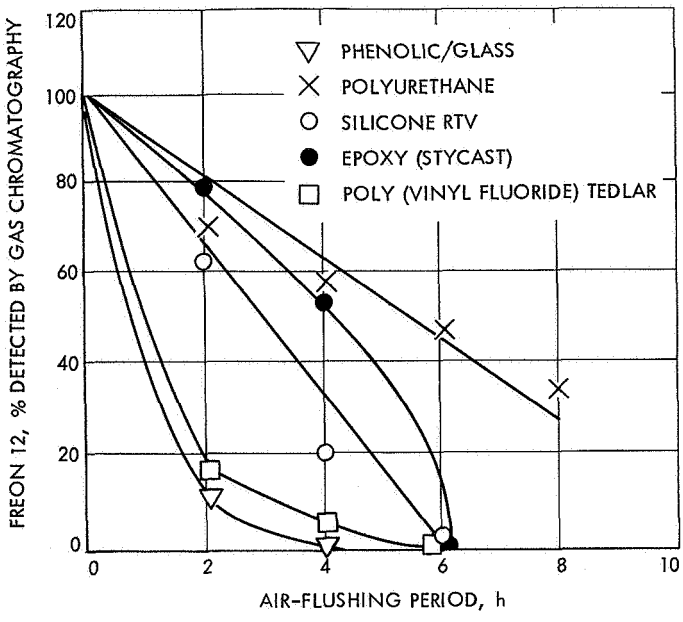

Fig. 4. Relationship of Freon 12 desorption with air-flush time

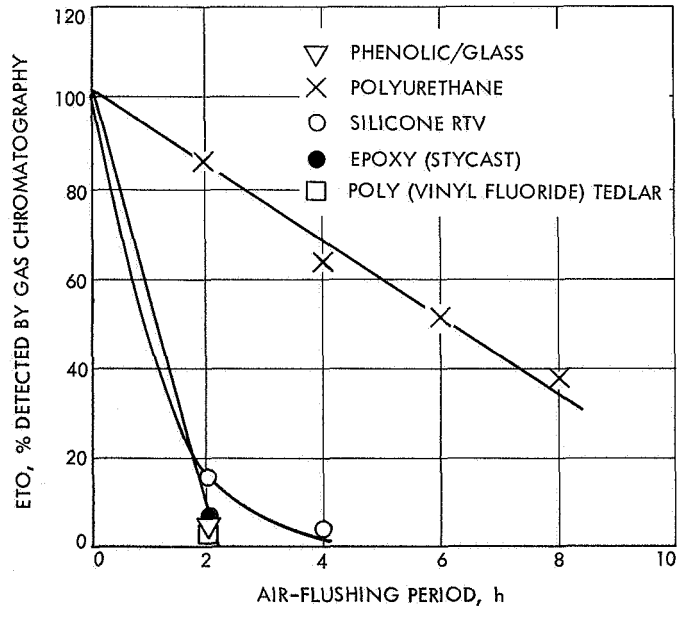

Fig. 5. Relationship of ETO desorption with air-flush time

3. Conclusion. The conclusion reached from these experiments is that flush time should be extended. Complete desorption at ambient pressure from polymeric materials may take days; however, the results of these experiments indicate that 90 to 100% of the absorbed sterilant gases could be expelled from most of the materials tested after 8 h of air flushing.

D. Determination of Moisture Content

The rate of sterilization with ETO is dependent, among other factors, on the moisture content or relative humidity (RH) in the decontamination chamber. It has been found that ETO decontamination is most rapid at 30 to 35% RH (Ref. 8). The sporicidal action decreases as the RH either increases or decreases from this value. The adjustment of RH in the chamber, and its continuous monitoring is, therefore, very important.

The investigations carried out concerning humidity determinations consisted of the evaluation of commercially available humidity-sensing instruments. A literature search indicated that very little work was done with respect to moisture determination in atmospheres other than air or inert gases (Refs. 9 and 10). Instruments available were developed mainly for the determination of moisture in air. Some of these instruments had the promise of satisfactorily fulfilling the following requirements for moisture determination in an ETO–Freon 12 environment:

(1) The sensor probe must remain unaffected by ETO–Freon 12 for long periods at a moderately high, 50°C, temperature.

(2) The instrument must be capable of remote reading of moisture content in the decontamination chamber.

(3) The device must be accurate and give repeatable results.

(4) The cost and operation of the instrument must be inexpensive.

The present evaluation was comparative in nature. As a reference standard, an Alnor Model 7000 U[3] (described later) was used. This instrument is considered to be capable of measuring RH with an accuracy of ±2%.

1. Definition of terms. The definition of certain terms used in moisture measurements will be useful at this time:

(1) *Dry-bulb temperature* is the temperature of the gas indicated by a thermometer.

(2) *Wet-bulb temperature* is the temperature as read by a thermometer, the bulb of which is covered with a distilled-water-wetted cloth sleeve. A temperature depression results from the evaporative cooling effect.

[3]Manufactured by Illinois Testing Laboratories, Chicago, Illinois.

(3) *Relative humidity* is the ratio of the actual vapor pressure in the mixture to the saturated vapor pressure, with respect to water at the prevailing dry-bulb temperature. It is expressed in percent.

(4) *Dew point* is that unique temperature to which any gas must be cooled to be saturated with respect to water. It is expressed in degrees Celsius or Fahrenheit.

2. *Selection of instruments for evaluation.* The widespread need for accurate humidity control and monitoring has resulted in the development of many basic types of instruments. The types commercially available at the present can be classified as follows:

(1) Natural fiber (Refs. 11 and 12)

(2) Wet-and-dry bulb (Ref. 13)

(3) Hygroscopic salt, Dunmore type (Ref. 14)

(4) Electrical resistance or polyelectrolyte type (Ref. 15)

(5) Electrical impedance (Ref. 16)

(6) Manual dew-point meter (Ref. 17)

(7) Cold-mirror optical dewpointer (Ref. 18)

(8) Other: Infrared spectrophotometer, thermal conductivity meter (Ref. 10)

Among these the ones considered suitable for evaluation in an atmosphere of ETO–Freon 12 were: the electrical resistance type, the electrical impedance type, and the cold-mirror optical dew-pointer. The others were considered unsuitable for service in the environment of interest.

In addition to the Alnor instrument which served as a standard, a wet-and-dry bulb psychrometer was used for the comparison of data.

3. *Description of instruments.*

a. Electrical resistance type (El-Tronics Model 102)[4] (Ref. 19). This instrument consists of an electric circuit capable of measuring the resistance of a proprietary wafer, made of sulfonated polystyrene, with a pair of gold electrodes deposited on the surfaces. As the wafer adsorbs moisture, the surface resistance changes in the characteristic manner illustrated in Fig. 6. The output is read directly as percent relative humidity, on a meter. Periodic calibration can be obtained by substituting a known resistance in place of the sensor element and adjusting the meter to full scale. The wafer element is illustrated in Fig. 7. A temperature calibration chart is provided with the instrument.

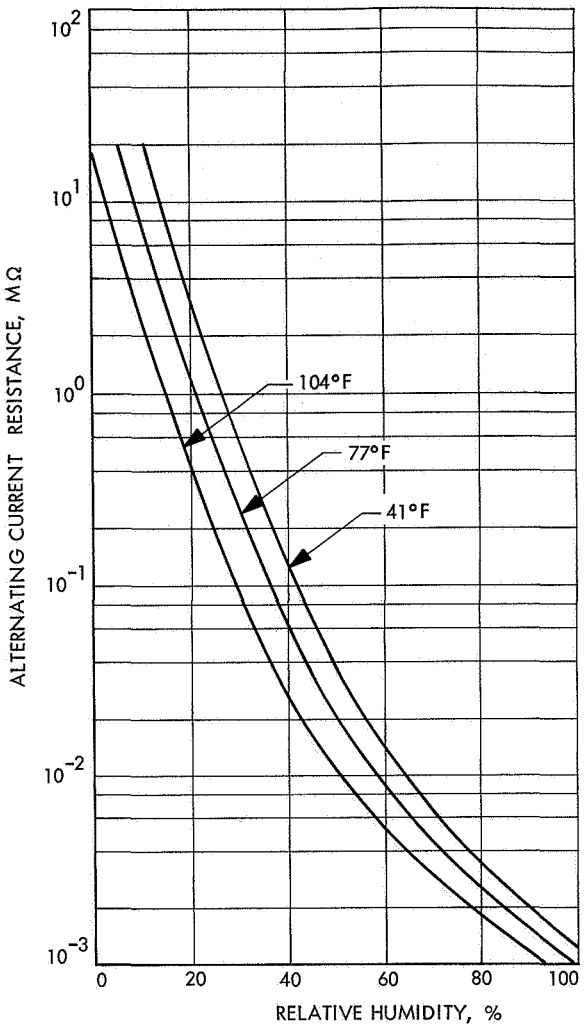

Fig. 6. Relationship of relative humidity and resistance for El-Tronics humidity sensing element

b. Electrical impedance type (Panametrics Model 1000).[5] The operation of this sensor (Ref. 20) depends on the impedance of a thin layer of aluminum oxide placed in between pure aluminum and gold faces. By means of a suitable measuring circuit (Fig. 8), the electrical impedance of the sensor is displayed on a meter, the value of which can be converted to the dew point using a calibration curve, supplied by the manufacturer. An illustration of the basic element, known as the Stover type, is

[4]Manufactured by El-Tronics, Warren, Pennsylvania.

[5]Manufactured by Panametrics, Inc., Waltham, Massachusetts.

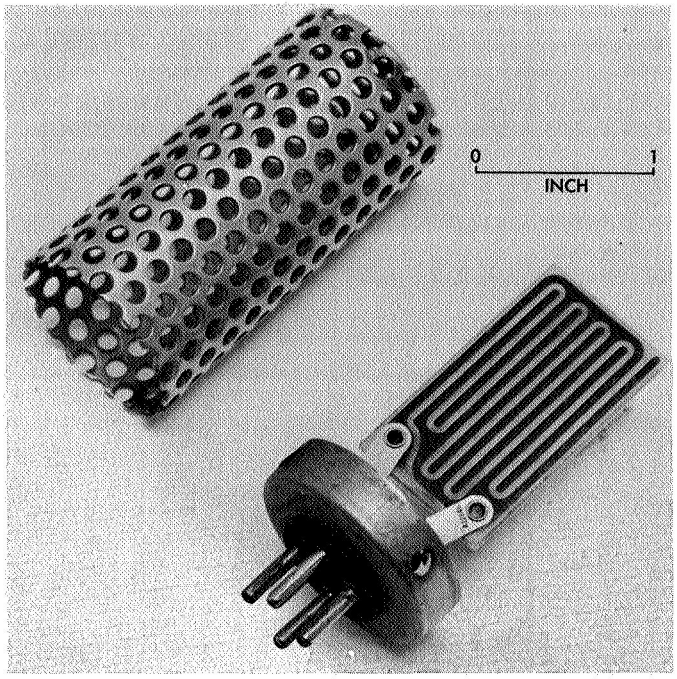

Fig. 7. El-Tronics sensing element

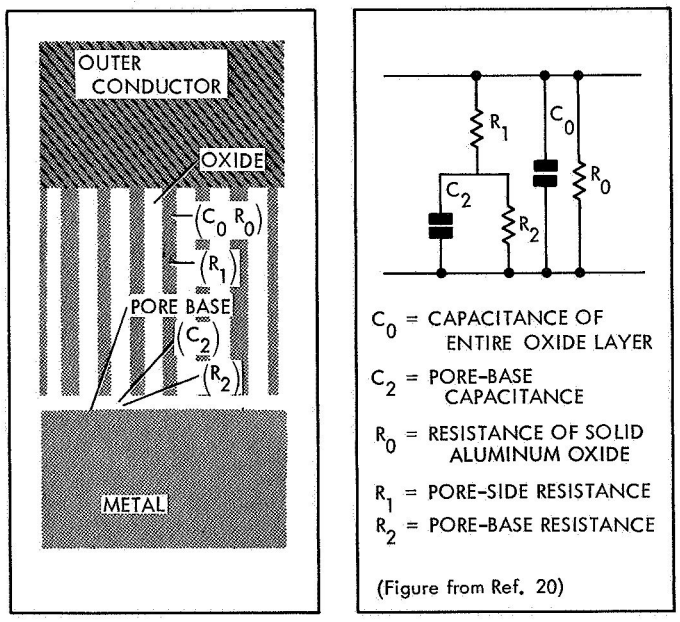

Fig. 8. Panametrics sensor; equivalent circuit of a pore

illustrated in Fig. 9. The electrical behavior of this type of element may be described in terms of a single pore of aluminum oxide and an equivalent electrical circuit for that pore (Fig. 8).

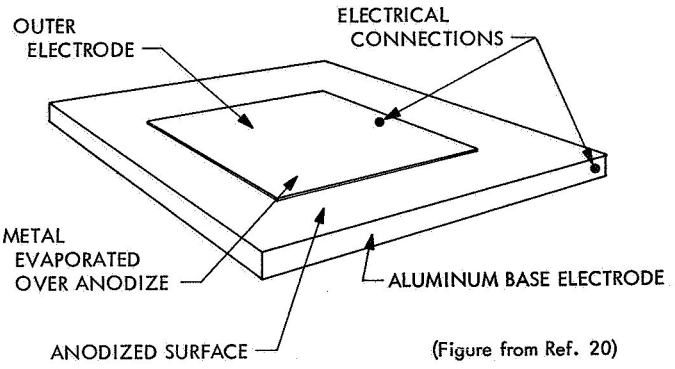

Fig. 9. Stover-type aluminum oxide humidity element

The following equation shows the functional relationship of impedance to other parameters:

$$Z = g\left\{\frac{A}{8\pi^2 f^2} \pm \frac{1}{2}\left[\left(\frac{A}{4\pi^2 f^2}\right)^2 - \frac{1}{\pi^2 f^2 R_p^2}\right]^{1/2}\right\} \qquad (2)$$

where

Z = impedance, Ω

f = voltage frequency, Hz

R_2 = leakage resistance of Al_2O_3, Ω

R_p = equivalent resistance of Al_2O_3, Ω

C_2 = capacitance of Al_2O_3, F

$$A = 4\pi^2 f^2 C_2 + \frac{1}{R_2^2 C_2}$$

c. Cold-mirror optical dew-point type (Technology-Versatronics, Model 707)[6] (Refs. 18–20). This instrument utilizes an optical-sensing technique, a thermoelectric element and a linear thermometer (Figs. 10 and 11). The optical technique detects the formation of dew on the sensor mirror. The temperature is monitored by the linear thermometer, the output of which is displayed as a direct dew-point reading on a meter. The cooling of the sensor mirror depends on the bismuth telluride semiconductor Peltier effect. The more important properties of this semiconductor are expressed by a figure of merit, Z, as follows:

$$Z = g\left(\frac{\pi^2}{T_c^2 \rho K}\right) \qquad (3)$$

[6]Manufactured by Technology-Versatronics, Yellow Springs, Ohio.

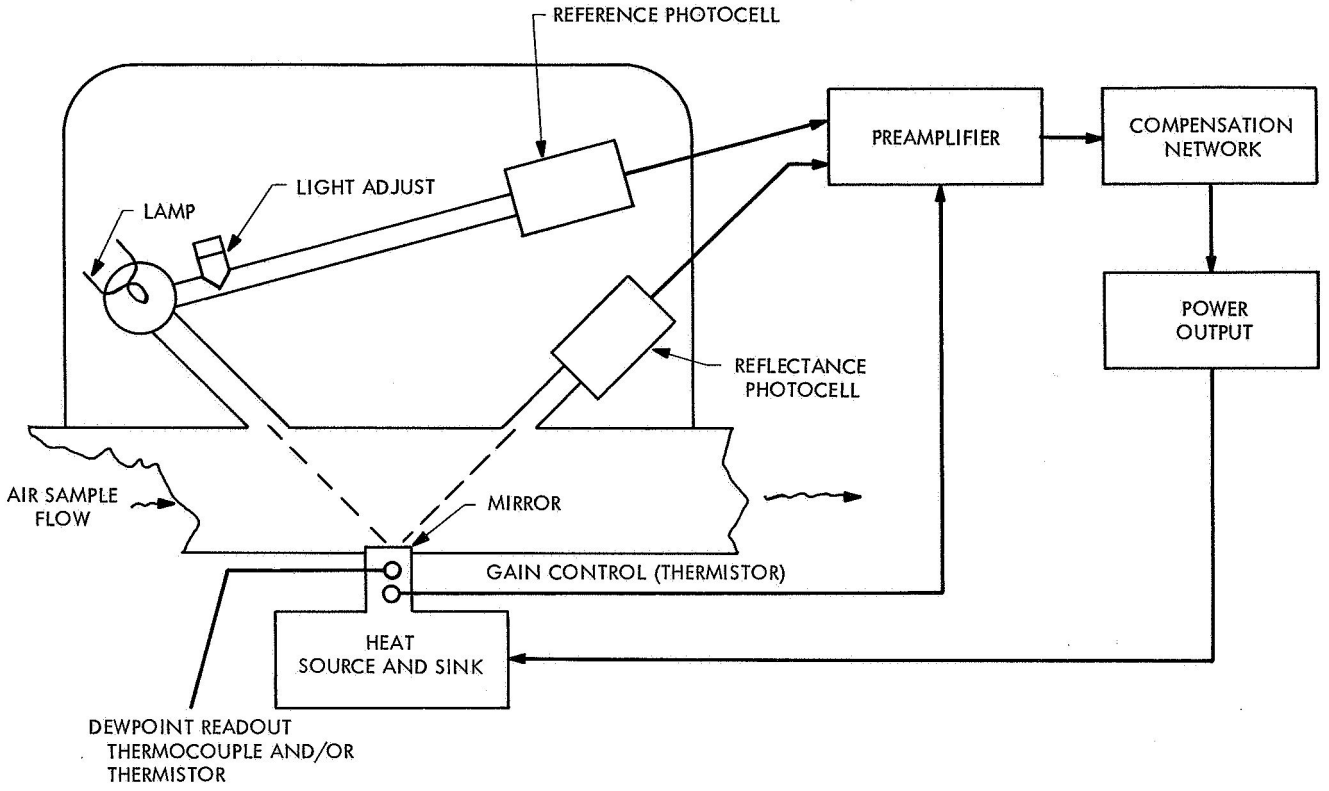

Fig. 10. Functional block diagram for a typical cold-mirror hygrometer

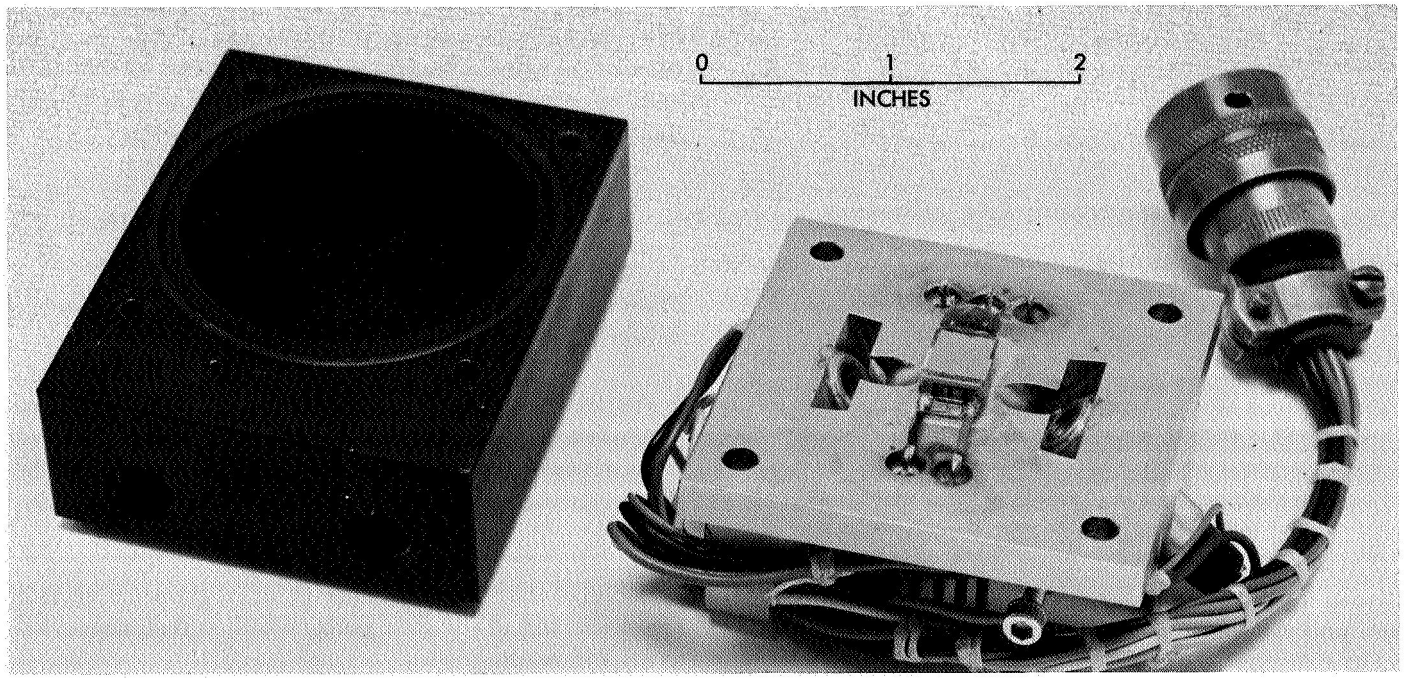

Fig. 11. Technology-Versatronics cold-mirror-type humidity sensor

where

π = the Peltier coefficient

T_c = the cold face temperature

ρ = the electrical resistivity

K = the thermal conductivity

For good refrigeration, Z should be maximized; therefore, ρ and K should have values as low as possible and π should be as high as possible. Best Z values currently obtainable from commercial materials is about

$$3 \times 10^{-3}\ ^{\circ}C^{-1}$$

d. Manual dew-point type (Alnor, Model 7000U).[7] In the operation of the Alnor Dewpointer (Ref. 21), the gas mixture is drawn into the dewpointer observation chamber at a pressure above that of the atmosphere. A gauge indicates directly the ratio between the pressure of the gas sample and that of the atmosphere. An operating valve is then depressed, allowing the gas sample in the observation chamber to expand rapidly to atmospheric pressure. Meantime, a lamp illuminates the chamber. If the gas, due to its rapid expansion, has cooled below its dew point, a distinctive fog is observed in the chamber, through a lens system. This procedure is repeated to find the pressure ratio at the time the fog starts to vanish. The dew point of the gas sample may then be determined from the following equation:

$$T_{dewpoint} = (T_i + 460)\,(PR)^{Q} - 460 \qquad (4)$$

where

T_i = initial temperature of the gas sample

PR = pressure ratio

$$Q = \frac{K-1}{K}$$

$= 0.2855$ for air

and

$$K = \frac{C_p}{C_v}$$

C_p and C_v are specific heats of the gas at constant pressure and constant volume, respectively.

[7]Manufactured by Alnor Division of Illinois Testing Laboratories, Chicago, Illinois.

e. Wet-and-dry bulb psychrometer (Bendix Model 566).[8] In this instrument (Ref. 13), a battery-operated fan causes the ambient air to circulate uniformly over both the wet and dry bulbs at an optimal air velocity for the instrument. Readings of both wet and dry bulbs are taken after the instrument has reached a state of equilibrium. Averages of several readings are recorded.

4. Data reduction. With the exception of the electrical resistance type (El-Tronics), all other instruments give readings in some parameter other than RH. The data obtained have to be converted to RH values to facilitate the comparative evaluation.

Raw data from the wet-and-dry thermometry consists merely of the temperatures of the wet and dry bulbs. The values of RH, which are obtained from psychrometric tables, are based on the Ferrel equation (Ref. 22):

$$e = e' - 0.000367\,P\,(t - t')\left(1 + \frac{t' - 32}{1571}\right) \qquad (5)$$

where, t and t' are the temperatures of the dry and wet bulb thermometers, P is the barometric pressure of air in inches, e' is the saturation pressure of water vapor at t'. Solution of the Ferrel equation gives e, the vapor pressure of water at t.

Readings from the Alnor dew-point instrument consist of the pressure ratio and the ambient temperature. The dew point can be calculated by the use of Eq. (3). Such calculation is unnecessary, however, because of the existence of conversion tables. The RH can be obtained from the dew point using the following relationship:

$$\frac{\text{vapor pressure at dew point} \times 100}{\text{vapor pressure at room temperature}} = \%\,\text{RH}$$

The use of the Smithsonian Tables (Ref. 23) facilitates the data reduction.

For the electrical impedance type of instrument, the meter reading is converted to the dew point by use of the calibration curve supplied by the manufacturer. The dew point is converted to the RH value as indicated above.

The cold-mirror optical dew-point hygrometer gives a direct reading of the dew point, which is then converted to an RH reading.

[8]Manufactured by Bendix Instruments, Inc.

The electrical-resistance-type instrument gives a direct reading in % RH. No data reduction is necessary.

5. Test procedure. The procedure followed in testing the instruments is shown diagramatically in Fig. 12. For the first exposure conditions, namely at room conditions in air, readings were taken twice a day for one week with all the instruments. At least five readings were taken at each of the other exposure conditions with each instrument. Not all the instruments were usable at some of the conditions. For example the wet-and-dry bulb thermometer could not be used in the warm chamber or in the ETO–Freon 12 chamber. The Alnor dewpointer could not be used in the ETO–Freon 12 atmosphere.

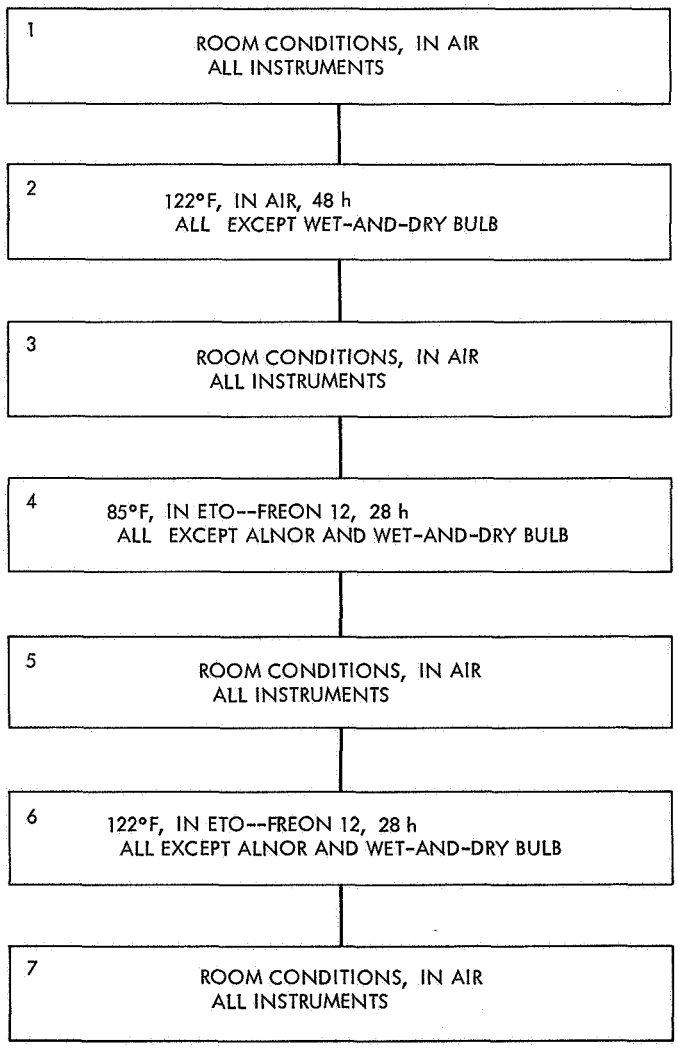

Fig. 12. Test sequence for the evaluation of moisture detectors

6. Results and discussion. The initial relative humidity readings in air obtained from the reference instrument, the Alnor dewpointer, and from the test instruments (Panametrics, El-Tronics and Technology-Versatronics) differed significantly. Variations up to 4 percentage points in RH were observed. The disagreement between the test instruments themselves was also significant. These are shown in Fig. 13. Values obtained by the Bendix wet-and-dry thermometer are also included in this figure.

The tendency of the Technology-Versatronics was to read higher RH values, and the tendency of the Panametrics and El-Tronics was to register lower RH values, as compared with the Alnor.

Significant changes occurred in the Panametrics and the Technology-Versatronics probes when they were exposed to ETO–Freon 12 either at room temperature or at 50°C. Under these conditions, readings from the Panametrics were exceedingly low, and from the Technology-

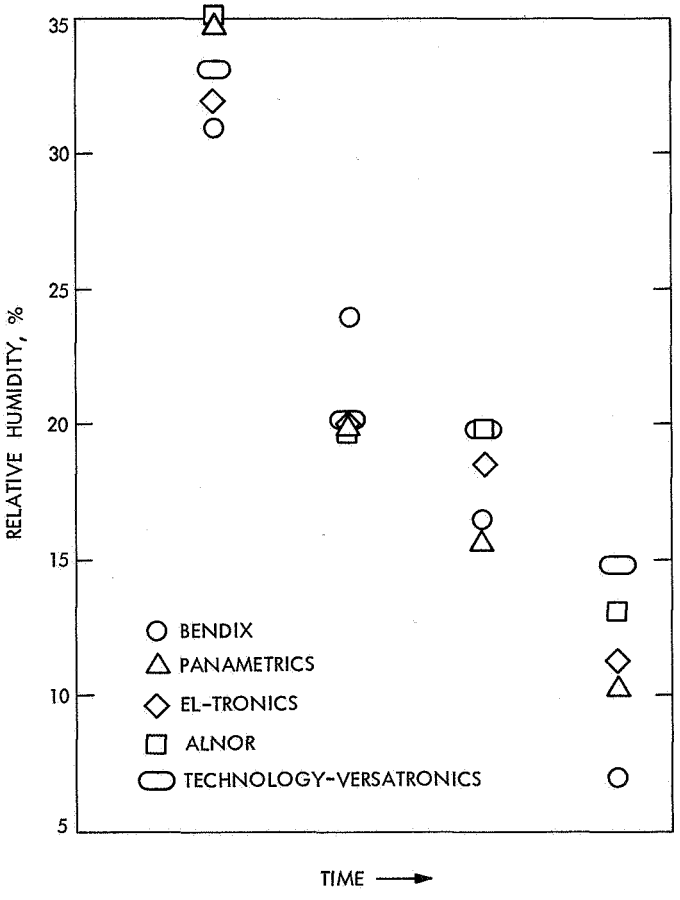

Fig. 13. Readings for various humidity sensing instruments at room conditions

Versatronics, they were quite high. Bringing the probes to atmospheric conditions did not change the situation.

A vacuum treatment of the Panametrics probe, to dissipate any absorbed sterilant gas did not improve its operation. Likewise, repeated cleaning of the Technology-Versatronics mirror did not bring about a recovery. Indications were that the probes of both instruments were irreversibly affected by the ETO–Freon 12 gas mixture. It is thought that both the Al_2O_3 of the Panametrics probe and the bismuth telluride semiconductor of the Technology-Versatronics probe reacted with the ETO to change their capacitance or resistance.

The El-Tronics probe was the least affected. Readings after 1 cycle of ETO–Freon 12 exposure were, however, much lower with reference to the Alnor than they were before exposure. Whereas, before exposure, % RH readings were in the order of 1 to 3 percentage points below those of the Alnor readings, they had increased 8 to 10 points after exposure (Figs. 13 and 14). Washing the El-Tronics sensor element with distilled water improved its sensitivity (Fig. 15). Further exposure to ETO–Freon 12 cycles once again changed its sensitivity; but there was no indication of a continuous deterioration of the element as it was exposed to a second, third, fourth, fifth, and sixth cycles of ETO–Freon 12 decontamination. Figure 16 shows the % RH readings from the Alnor and the El-Tronics sensor after the sixth cycle. A second washing with water at this point did not improve the sensor's performance.

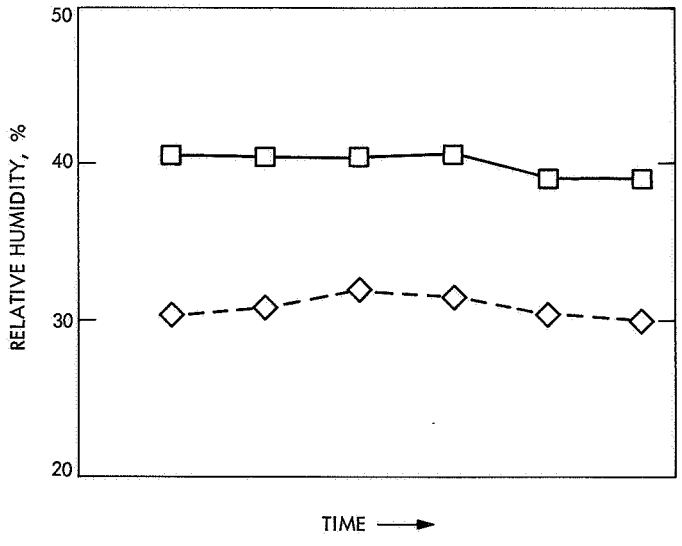

Fig. 14. Humidity readings of El-Tronics after 1 cycle of exposure to ETO–Freon 12

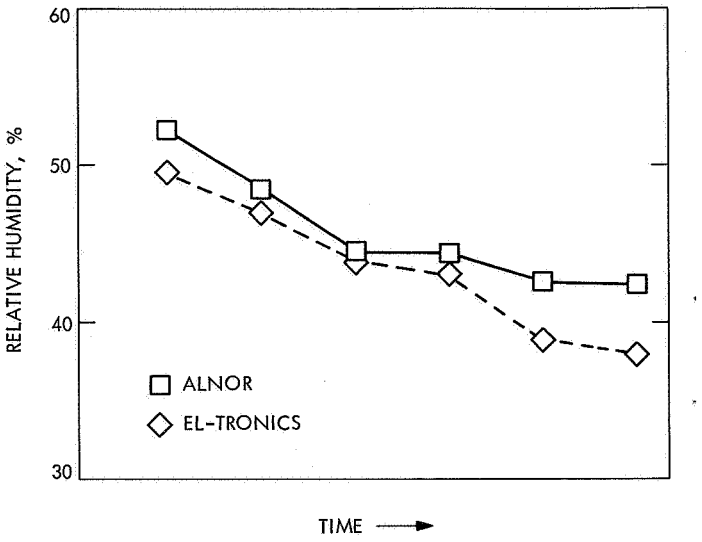

Fig. 15. Humidity readings of El-Tronics after washing with distilled water

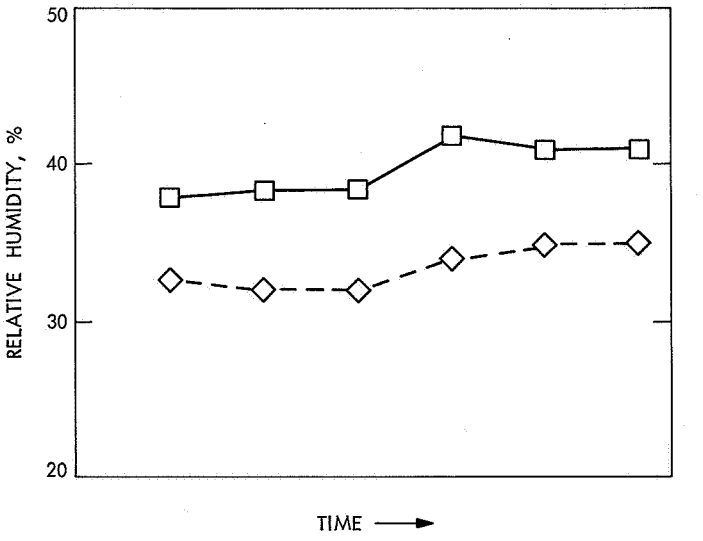

Fig. 16. Humidity readings of El-Tronics after 6 cycles of exposure to ETO–Freon 12

7. *Conclusions.* The humidity sensors evaluated are not suitable for moisture determination in an atmosphere of ETO–Freon 12. One of these, the El-Tronics electric-resistance-type sensor is apparently far better than the others. This sensor, after exposure to ETO–Freon 12, tends to give lower % RH values than the actual, as determined by the reference standard. A need exists for the adequate means of determining moisture in the atmosphere of the ETO decontamination chamber.

III. Effects of Various Sterilization Cycles on Selected Polymeric Products

A. Introduction

Sterilization is a time- and temperature-dependent variable. It was of interest to find out what effects various heat cycles would have on the properties of polymeric products.

Sterilization cycles are based on the destruction of different spore-forming bacteria. One of the most heat-resistant types is the indigenous mesophilic spore found in soil. Spacecraft sterilization cycles are based on the D values (decimal destruction time) of these spores. The D value is defined as the time necessary, at any given temperature, to kill 1 log value or 90% of the spore population. The equivalent sterilization time, F_T, at any given temperature is:

$$F_T = D_T \log \frac{N_0}{P_s} \qquad (6)$$

where

N_0 = the initial number of microorganisms

P_s = probability of survival

For a D value of 1.8 h at 135°C, an initial population of 10^8 spores and a probability of survival of 1 in 10,000 (or 10^{-4}), the equivalent sterilization time will be:

$$1.8 \times \log \frac{10^8}{10^{-4}} = 1.8 \times 12 = 22 \text{ h}$$

The equivalent sterilization times, F_T, at different temperatures for indigenous mesophilic spores in soil, as well as for two other spore formers, are given in Table 2 (also see Ref. 8).

For the type-approval testing of such spacecraft components and materials as polymeric products, the dry-heat sterilization cycles are extended considerably. At 135°C, cycles are 92 h, and six such cycles are specified. These test conditions are far more rigorous than the biological requirements, and are set to meet a number of possible needs—for example, the need for resterilization after repairs and the need for complete assurance of the sterilizability of components and subassemblies.

The acceptable sterilization temperatures range from 105°C to 160°C (Ref. 24). For this study, the two temperatures chosen were (1) the lowest of this range, 105°C, and (2) one close to the top of the range, 155°C. The cycles were as follows:

40 h at 155°C

600 h at 105°C

These are considered *biologically equivalent* to the specified cycle of 92 h at 135°C. The products were exposed six times to each of the above cycles.

Table 2. Comparison of sterilization cycles based on destruction of different sporeformers in various environments (cycles based on 12 logarithms (12D) of spore reduction)

Temperature, °C	Sterilization time F_T, h		
	B. subtilis var. *niger*, on paper strip[a]	*B. subtilis* var. *niger*, in solids[a]	Indigenous mesophilic spores, in soil[b]
105	59.0	228.0	336.0
110	35.0	144.0	210.0
115	20.0	84.0	132.0
120	12.0	54.0	84.0
125	6.7	32.0	53.0
130	4.0	20.0	34.0
135	2.3	12.0	22.0
140	1.3	7.5	14.0
145	0.65	4.6	8.8
150	0.43	2.8	5.5
155	0.25	1.7	3.7
160	0.14	1.1	2.5

[a]Recovered in trypticase soy broth.

[b]Recovered in thioglycolate broth.

B. Experimental

1. Materials and preparation of samples for testing. The six products selected for this investigation were materials that had been tested previously and had shown sterilizability. Each of the products represented a functional category, as follows:

Epon 934	adhesive
Stycast 1090	encapsulant
Viton	elastomeric gasket
EG 758	reinforced plastic
Tedlar	film
Mystik 7352	adhesive tape

The above products were obtained from the manufacturers, ready for use, except for Epon 934 and Stycast 1090, which needed preliminary handling and casting before test specimens could be prepared from them. All test samples were prepared in accordance with the sizes and shapes specified in the pertinent standard test methods.

2. Test equipment and procedures. The ETO decontamination setup and the thermal sterilization chambers used were described previously (Refs. 4 and 9; see also Ref. 25). Other test equipment or apparatus used were standard and, therefore, need no description.

The ETO–Freon decontamination, as well as the dry-heat sterilization procedures, used were in conformance with the JPL type approval procedures, described before. The performance of the thermal sterilization chambers was evaluated by mass spectrographic analysis of the chamber atmosphere at the beginning and near the end of a cycle, with or without samples present. The maximum amount of oxygen detected in these nitrogen-filled ovens at the start of a cycle was 0.01%, and it increased to 0.02% near the end of the cycle, when no samples were present. With samples present, the maximum amount of oxygen detected near the end of a cycle was 0.03%. This increase is partly ascribable to desorbed air from the samples.

For the evaluation of the effects of an atmosphere of nitrogen containing small percentages of oxygen on polymeric products, a specially prepared mixture containing 0.45% O_2 and 99.55% N_2, by weight, was used. The test chamber was filled with this mixture after repeated evacuation and purging. A steady flow of the mixture was maintained during each cycle, at a rate of 10 ml/min.

3. Tests used. For each class of product, the tests and methods used are listed in Table 3. Testing was performed in full compliance with the standard methods.

C. Results and Discussion

Test results for the six polymeric products before and after exposure to various conditions are summarized in Tables 4 to 9. Detailed results are given in Appendix B. Changes in the properties of the products after various exposure conditions are given in Tables 10 to 15.

Table 3. Evaluation tests

Test	Adhesive	Elastomer	Encapsulant	Film sheet	Plastic laminate	Tape	Standard
Hardness	X	—	X	—	X	—	ASTM D2240-64T ASTM D676-59T ASTM D785-62
Tensile strength	—	X	—	X	X	—	ASTM D412-62T and 64T ASTM D882-61T ASTM D638-61T
Elongation	—	X	—	X	X	X	ASTM D412-62T ASMT D88-61T ASMT D638-61T ASTM D1000-64
Tear	—	—	—	X	—	—	ASTM D624-54
Tensile shear strength	X	—	—	—	—	—	FTMS 175-Method 1033-1T
Adhesion (peel, scrape)	X	—	—	—	—	X	ASTM D1876-61T ASTM D2197-65T
Breaking strength	—	—	—	—	—	X	ASTM D1000-64
Compression	—	X	—	—	—	—	ASTM D395-61, Method B
Volume resistivity	—	—	X	X	X	X	ASTM D257-61T
Dielectric strength	—	—	X	X	X	X	ASTM D257-61T
Volume change	—	X	X	X	X	X	Direct measurement using an Ames dial gage micrometer
Weight loss	X	X	X	X	X	X	Direct weight measurement using a Mettler model H15 balance

Table 4. Summary of test results for ETO decontamination and dry-heat sterilization under various conditions for adhesive Epon 934

Commercial designation	Material type	Exposure conditions	Mechanical properties			Thermal property
			Hardness, Shore D	Shear strength, psi	T-peel strength, pi width	Weight change, %
Epon 934 A/B,[a]	Epoxy	Control	76.9	576	45.3	
		ETO	80.7	504	20.9	+0.280
		ETO + thermal (6 × 40 h at 155°C in N_2)	80.9	835	25.0	−0.306
		ETO + thermal (6 × 92 h at 135°C in N_2 + 45% O_2)	81.8	1170	35.2	−0.168
		ETO + thermal (6 × 92 h at 135°C in N_2)	79.3	1970	34.0	−0.097
		ETO + thermal (6 × 600 h at 105°C in N_2)	80	977	23	−0.029

[a]Manufacturer: Shell Chemical Company.

Table 5. Summary of test results for ETO decontamination and dry-heat sterilization under various conditions for elastomer Viton 377-9

Commercial designation	Material type	Exposure conditions	Mechanical properties						Physical and thermal properties	
			Hardness, Shore A	Tensile bars		Rings		Compression set, %	Volume change, %	Weight change, %
				Tensile strength, psi	Elongation, %	Tensile strength, psi	Elongation, %			
Viton 377-9,[a]	Fluoroelastomer	Control	82.6	2220	133	2290	165			
		ETO	79.0	1840	143	2200	176		+1.661	+1.061
		ETO + thermal (6 × 40 h at 155°C in N_2)	82.3	1850	110	2050	105	80.19	−0.395	+0.026
		ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	83.8	1890	110	2070	146	65.77	−0.370	+0.035
		ETO + thermal (6 × 92 h at 135°C in N_2)	82.2	1780	110	2250	138	73.10	−0.039	−0.081
		ETO + thermal (6 × 600 h at 105°C in N_2 + 0.45% O_2)	81	2220	120	2480	209	68.4	+0.92	+0.111

[a]Manufacturer: (DuPont material as used in test) Parker Seal Company.

Table 6. Summary of test results for ETO decontamination and dry-heat sterilization under various conditions for encapsulant Stycast 1090

Commercial designation	Material type	Exposure conditions	Mechanical properties	Electrical properties		Physical and thermal properties	
			Hardness, Shore D	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
Stycast[a] 1090/ Catalyst 11	Epoxy resin	Control	71.8	1.38×10^{14}	434		
		ETO	70.6	7.2×10^{13}	381		+0.291
		ETO + thermal (6 × 40 h at 155°C in N_2)	76.9	1.85×10^{14}	286	−0.319	−1.249
		ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	75.1	1.76×10^{14}	327	−0.175	−0.786
		ETO + thermal (6 × 92 h at 135°C in N_2)	77	2.1×10^{14}	—	−0.571	−0.626
		ETO + thermal (6 × 600 h at 105°C in N_2)	76	9.4×10^{13}	337	−0.343	−0.263

[a]Manufacturer: Emerson and Cuming, Inc.

Table 7. Summary of test results for ETO decontamination and dry-heat sterilization under various conditions for film Tedlar, type 30

Commercial designation	Material type	Exposure conditions	Mechanical properties			Electrical properties		Physical and thermal properties	
			Tensile strength, psi	Elongation, % (in 1.0 in.)	Tear strength, lb/mil	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
Tedlar,[a] type 30, modification B, high gloss, 1 mil	Poly (vinyl fluoride)	Control	11800	134	1.83	1.6×10^{15}	3280		
		ETO	10500	125	1.27	2.5×10^{15}	3510	−0.816	+0.474
		ETO + thermal (6 × 40 h at 155°C in N_2)	9520	98	1.28	1.4×10^{15}	3580	−9.175	−0.154
		ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	10100	92	1.38	1.29×10^{15}	3660	−5.012	+0.014
		ETO + thermal (6 × 92 h at 135°C in N_2)	11220	85	1.26	2.7×10^{15}	3600	−4.518	+0.042
		ETO + thermal (6 × 600 h at 105°C in N_2)	10900	123	1.23	1.27×10^{15}	3450	−3.254	−0.740

[a]Manufacturer: DuPont.

Table 8. Summary of test results for ETO decontamination and dry-heat sterilization under various conditions for plastic laminate EG 758

Commercial designation	Material type	Exposure conditions	Mechanical properties			Electrical properties		Physical and thermal properties	
			Hardness, Rockwell E	Tensile strength, psi	Elongation, %	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
EG 758,[a] type GEE	Epoxy/glass	Control	86.9	40300	3.35	3.4×10^{15}	> 383		
		ETO	87.7	47300	2.13	4.6×10^{15}	> 387	0	+0.053
		ETO + thermal (6 × 40 h at 155°C in N_2)	86.1	46800	2.10	1.28×10^{16}	> 391		−0.256
		ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	89.2	46900	1.68	7.2×10^{15}	> 374	−0.146	−0.178
		ETO + thermal (6 × 92 h at 135°C in N_2)	90.4	39800	1.43	1.25×10^{16}	> 376	+0.042	−0.202
		ETO + thermal (6 × 600 h at 105°C in N_2)	90	49400	1.43	3.9×10^{15}	376	−0.105	−0.102

[a]Manufacturer: Mica Corporation.

Table 9. Summary of test results for ETO decontamination and dry-heat sterilization under various conditions for tape Mystik 7352

Commercial designation	Material type	Exposure conditions	Mechanical properties			Electrical properties		Physical and thermal properties	
			Peel adhesion oz/in. width	Breaking strength, lb/in.	Elongation, %	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
Mystik 7352[a]	Polyester	Control	44.1	49	70	1.83×10^{15}	1980		
		ETO	43.6	45	52	1.32×10^{15}	1835	0	+0.772
		ETO + thermal (6 × 40 h at 155°C in N_2)	90.4	43	54	1.35×10^{16}	1780	−1.962	−2.108
		ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	99.2	41	55	8.4×10^{15}	1887	−0.910	−1.511
		ETO + thermal (6 × 92 h at 135°C in N_2)	—	38	56	2.4×10^{16}	1870	−2.256	−1.194
		ETO + thermal (6 × 600 h at 105°C in N_2)	79.2	46	66	7.9×10^{15}	1800	−0.316	−1.595

[a]Manufacturer: Mystik Tape, Inc., Division of the Borden Chemical Company.

Table 10. Changes in properties of adhesive Epon 934 after ETO decontamination and dry-heat sterilization

Product and material type	Exposure conditions	Unit change in hardness	Shear strength, % retained	T-peel strength, % retained	Weight change, %	Compatibility rating
Epon 934 A/B	ETO	+3.9	87.5	46.1	+0.280	—
	ETO + thermal (6 × 40 h at 155°C in N_2)	+4.1	145.0	55.2	−0.306	C
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	+4.9	203.1	77.7	−0.168	C
	Thermal (6 × 92 h at 135°C in N_2)	+2.4	342.0	75.0	−0.097	C
	ETO + thermal (6 × 600 h at 105°C in N_2)	−0.9	170	50.8	−0.029	C

Table 11. Changes in properties of elastomer Viton 377-9 after ETO decontamination and dry-heat sterilization

Product and material type	Exposure conditions	Unit change in hardness	Bars		Rings		Compression set, %	Volume change, %	Weight change, %	Compatibility rating
			Tensile strength, % retained	Elongation, % retained	Tensile strength, % retained	Elongation, % retained				
Viton 377-9	ETO	−3.6	82.5	107.5	96	106.7		+1.661	+1.061	—
	ETO + thermal (6 × 40 h at 155°C in N_2)	−0.3	83.0	82.7	89.5	63.5	80.19	−0.395	+0.026	M
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	+1.2	84.8	82.7	90.4	88.5	65.77	−0.370	+0.035	C
	ETO + thermal (6 × 92 h at 135°C in N_2)	−0.4	80	82.7	98.3	83.6	73.10	−0.039	−0.081	C
	Thermal (6 × 600 h at 105°C in N_2)	−1.6	100	90.2	108.3	126.7	68.4	+0.92	+0.111	C

Table 12. Changes in properties of encapsulant Stycast 1090 after ETO decontamination and dry-heat sterilization

Product and material type	Exposure conditions	Unit change in hardness	Volume change, %	Weight change, %	Compatibility rating
Stycast 1090/Catalyst 11	ETO	−1.2	+0.057	+0.291	—
	ETO + thermal (6 × 40 h at 155°C in N_2)	+4.0	−0.319	−1.249	M
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	+3.3	−0.175	−0.786	C
	ETO + thermal (6 × 92 h at 135°C in N_2)	+5.2	−0.571	−0.626	C
	ETO + thermal (6 × 600 h at 105°C in N_2)	+4.2	−0.343	−0.263	C

Table 13. Changes in properties of film Tedlar 30 after ETO decontamination and dry-heat sterilization

Product and material type	Exposure conditions	Tensile strength, % retained	Elongation, % retained	Tear strength, % retained	Volume change, %	Weight change, %	Compatibility rating
Tedlar, type 30, modification B, high gloss, 1 mil	ETO	89.0	93.3	69.4	−0.816	+0.474	—
	ETO + thermal (6 × 40 h at 155°C in N_2)	80.7	73.1	70.0	−9.175	−0.154	NC
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	85.6	68.6	75.4	−5.012	+0.014	NC
	ETO + thermal (6 × 92 h at 135°C in N_2)	95	63.4	69	−4.518	+0.042	NC
	Thermal (6 × 600 h at 105°C in N_2)	92.4	91.7	67.2	−3.254	−0.740	NC

Table 14. Changes in properties of plastic laminate EG 758 after ETO decontamination and dry-heat sterilization

Product and material type	Exposure conditions	Units change in hardness	Tensile strength, % retained	Elongation, % retained	Volume change, %	Weight change, %	Compatibility rating
EG 758, type GEE	ETO	+0.8	117.4	63.6	0	+0.053	—
	ETO + thermal (6 × 40 h at 155°C in N_2)	−0.8	116.1	62.7	−0.095	−0.256	C
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	+2.3	116.4	50.1	−0.146	−0.178	C
	ETO + thermal (6 × 92 h at 135°C in N_2)	+3.5	98.7	42.7	+0.042	−0.202	C
	Thermal (6 × 600 h at 105°C in N_2)	+3.1	122.6	42.7	−0.105	−0.102	C

Table 15. Changes in properties of tape Mystik 7352 after ETO decontamination and dry-heat sterilization

Product and material type	Exposure conditions	Peel adhesion, % retained	Breaking strength, % retained	Elongation, % retained	Volume change, %	Weight change, %	Compatibility rating
Mystik 7352	ETO	98.8	91.8	74.3	0	+0.772	—
	ETO + thermal (6 × 40 h at 155°C in N_2)	205.0	87.8	77.1	−1.962	−2.108	M
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	224.9	83.7	78.6	−0.910	−1.511	M
	ETO + thermal (6 × 92 h at 135°C in N_2)	—	77.5	80	−2.256	−1.194	M
	Thermal (6 × 600 h at 105°C in N_2)	179.6	93.9	94.3	−0.316	−1.595	M

To evaluate the effects of various sterilization cycles or conditions on the materials, the changes in the properties after exposure were assessed and the products were rated compatible (C), marginal (M), and not compatible (NC), depending on the extent of change. The criteria used for rating have been discussed before (Refs. 1 and 4). In general, products were rated C when the retained mechanical and physical properties were 80% or more, and the weight loss was less than 1%. They were rated NC when less than 70% of the original properties were retained, and the weight loss was more than 4%. The M ratings were assigned whenever the values were in between those of C and NC. These criteria are restated in Appendix A, in more detail.

Table 16 shows the ratings assigned to each product at the various sterilization conditions. Since the number of products that were rated C is less after cycling at 155°C for 6 × 40 h, it can be concluded that sterilization under this condition is more harmful to a larger number of polymeric products than sterilization under the other conditions.

The ratings assigned for all the products are identical after exposure to the other conditions, namely 135°C for 6 × 92 h, with or without 0.45% oxygen present, and 105°C for 6 × 600 h. As far as the sterilizability of the products is concerned, these three conditions would have the same consequence.

Table 16. Compatibility ratings

Product	Rating after six sterilization cycles			
	40 h at 155°C in N_2	92 h at 135°C in 0.45% O_2 + N_2	92 h at 135°C in N_2	600 h at 105°C in N_2
Epon 934	C	C	C	C
Viton 377-9	M	C	C	C
Stycast 1090	M	C	C	C
Tedlar 30	NC	NC	NC	NC
EG 758	C	C	C	C
Mystik 7352	M	M	M	M

In attempting to evaluate the effects of various sterilization cycles on the properties of polymeric products, the method of compatibility rating is of limited usefulness. Ratings are based on criteria that define a wide range of values. For example, a marginal rating is assigned to products that lose 1 to 4% of their original weights. This does not distinguish a product that loses just a little more than 1% of its original weight from a compound that suffers almost four times the loss. Criteria for other properties define similar broad ranges of values. Moreover, products are rated M or NC, when there is one single failure in meeting any one criterion.

A better evaluation of the effects of various exposure cycles could be carried out if the real extent of property changes were considered and, instead of emphasizing one single property, the overall changes in properties were taken into account.

The data obtained were analyzed in the manner just stated. The properties of a given compound were first graded on the basis of the extent of change they underwent at a given sterilization condition; the higher the loss in property, the lower was the grade assigned. These grades were then averaged for each sterilization condition and compared with the other averages, to obtain a ranking for each sterilization condition. The method is shown in Table 17 for Viton 377-9.

It is seen in Table 17 that exposure at 155°C has the lowest average grade, and therefore, it is ranked 4th as a desirable sterilization cycle among the four investigated. Exposure at 105°C is ranked first; and there is little difference between the average grades for exposures at 135°C, with or without 0.45% oxygen present.

Similar treatment of the data obtained for the other products shows that sterilization at 155°C is also the least desirable for Epon 934, Tedlar 30, and Mystik Tape 7352 Stycast 1090 and EG 758 fare just as well or somewhat better at 155°C, as at the other conditions.

Besides Viton 377-9, Mystik Tape 7352 and Tedlar 30 show less overall loss in properties when sterilized at

Table 17. Evaluation of property changes by grading

Exposure condition	Hardness	Tensile strength	Elongation	Compression set	Weight change	Average grade	Rank
155°C (240 h)	98	83	83	70	90	85.0	4th
135°C (552 h) 0.45% O_2	92	85	83	85	91	87.2	2nd
135°C (552 h)	97	80	83	77	98	87.0	3rd
105°C (3600 h)	90	100	90	82	80	88.5	1st

105°C. Epon 934 bears itself just as well at 135°C and EG 758 fares equally well at 135° or 155°C.

D. Conclusions

The exposure of a limited number of polymeric products to various type-approval sterilization conditions indicates the following:

(1) At the exposure conditions of 155°C for 6 × 40 h, the properties of most of the products are more adversely affected.

(2) Heat-resistant compounds, such as the glass-reinforced epoxy plastic, EG 758, are not any more affected by exposure to higher temperatures for shorter periods than to lower temperatures for longer periods.

(3) In general, sterilization at 105°C for 6 × 600 h results in lesser loss of physical and mechanical properties of the products.

(4) The presence of small quantities of oxygen (0.45%) does not affect significantly the property changes.

It could be added that, although sterilization at higher temperatures results in increased loss in properties, this increase is not substantial.

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Appendix A

Criteria for Rating the Compatibility of Polymeric Materials Under Sterilization Conditions

Criteria for Rating the Compatibility of Polymeric Materials

The criteria used in this report varied from one class to another; however, those used for the electrical properties, weight loss, and mechanical properties were common to all.

I. Electrical Properties

Threshold values set for electrical values were the following:

Volume resistivity, 10^7 Ω-cm

Dielectric strength, 200 V/mil

Products were considered *compatible* (C) where (1) the two electrical measurements remained greater than the threshold values, (2) the decrease in volume resistivity was less than 10^3 Ω-cm, and (3) the loss in dielectric strength was no more than 25% of the original value.

Products were rated *not compatible* (NC) if any one of these criteria was not met.

Products with borderline values were rated *marginal* (M).

II. Mechanical Properties and Weight Loss

The criteria for mechanical properties (except hardness) and for weight loss that were applied to products after the various dry-heat sterilization cycles are tabulated as follows:

Rating	Property retention after sterilization cycle, %	
	Mechanical property	Weight loss
C	>80	<1
M	70 to 80	≧1, <4
NC	<70	>4

III. Other Properties

Criteria were also used for hardness, volume change, and compression set. These requirements appear in the following tabulation.

Material	Hardness		Volume		Compression	
	Drop, units	Rating	Change, %	Rating	Set, %	Rating
Elastomers	≦6	C	<4	C	>75	NC
	≧10	NC	4 to 6	M		
			>6	NC		
Reinforced plastics	≦8	C	<4	C	—	—
	≧12	NC	4 to 6	M		
			>6	NC		
Encapsulants	≦10	C	<4	C	—	—
	≧15	NC	4 to 6	M		
			>6	NC		

Appendix B

Complete Test Data on ETO and Dry-Heat Sterilization at Various Conditions for Selected Products

Table B-1. Complete test results after ETO decontamination and dry-heat sterilization under various conditions for adhesive Epon 934

Commercial designation	Cure schedule for unheated controls		Exposure conditions	Mechanical properties				Thermal properties
	Duration, h	Temperature, °F		Hardness, Shore D	Stressed dimensions, in.	Shear strength, psi	T-peel strength, pi width	Weight change, %
Epon 934 A/B	1	180	Control	74.5 79.4 76.8 (av) 76.9	1.00/0.99 1.00/0.99 1.01/0.99	465 657 605 (av) 576	49.5 41.0 (av) 45.3	
			ETO	83.3 79.8 79.1 (av) 80.7	1.00/1.01 1.00/1.01 1.00/1.00	556 495 460 (av) 504	22.8 20.0 20.0 (av) 20.9	+0.260 +0.262 +0.319 (av) +0.280
			ETO + thermal (6 × 40 h at 155°C in N_2)	82 81 79.7 (av) 80.9	1.00/1.00 1.00/1.00	870 800 (av) 835	37.1 18.0 20.0 (av) 25.0	−0.245 −0.411 −0.261 (av) −0.306
			ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	81.7 82.7 81 (av) 81.8	1.00/1.00 1.01/1.01 1.00/1.01	1310 1100 1110 (av) 1170	31.1 39.2 (av) 35.2	−0.151 −0.120 −0.232 (av) −0.168
			ETO + thermal (6 × 92 h at 135°C in N_2)	78 81 79 (av) 79.3	1.00/1.00 1.00/1.00 1.00/1.00	2070 1890 1950 (av) 1970	24 37 41 (av) 34	−0.058 −0.135 −0.097 (av) −0.097
			ETO + thermal (6 × 600 h at 105°C in N_2)	83 78 80 (av) 80	1.00/0.98 1.01/0.99 1.05/1.00	1330 800 800 (av) 977	25 27 18 (av) 23	−0.0135 −0.0556 −0.0190 (av) −0.0294

Commercial designation	Exposure conditions	Mechanical properties				
			Tensile bars			Rings
		Hardness, Shore A	Stressed dimensions, in.	Tensile strength, psi	Elongation, %	Stressed dimensions, in.
Viton 377-9	Control	82.5	0.137/0.36	2050	130	0.067/0.132
		81.8	0.131/0.36	2420	150	0.067/0.135
		83.6	0.133/0.36	2210	120	0.067/0.132
		(av) 82.6		(av) 2220	(av) 133	
	ETO	78.4	0.136/0.36	1980	150	0.067/0.133
		78.3	0.134/0.36	1660	130	0.067/0.131
		80.2	0.135/0.36	1890	150	0.067/0.134
		(av) 79.0		(av) 1840	(av) 143	
	ETO + thermal (6 × 40 h at 155°C in N_2)	80.3	0.134/0.36	1660	100	0.131/0.067
		83.1	0.136/0.36	1920	110	0.134/0.067
		83.3	0.135/0.36	1980	120	0.134/0.067
		(av) 82.3		(av) 1850	(av) 110	
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	85.7	0.135/0.36	1850	110	0.132/0.067
		84.7	0.132/0.36	2020	110	0.129/0.066
		81	0.129/0.36	1790	110	0.135/0.067
		(av) 83.8		(av) 1890	(av) 110	
	ETO + thermal (6 × 92 h at 135°C in N_2)	82.7				0.132/0.067
		81.7	0.132/0.48			0.127/0.067
		82.3	0.131/0.48	1780	110	0.133/0.067
		(av) 82.2		(av) 1780	(av) 110	
	ETO + thermal (6 × 600 h at 105°C in N_2)	81	0.137/0.36	2330	120	0.131/0.068
		81	0.135/0.36	1930	120	0.131/0.068
		81	0.138/0.36	2410	120	0.131/0.068
		(av) 81		(av) 2220	(av) 120	

Table B-2. Complete test results after ETO decontamination and dry-heat sterilization under various conditions for elastomer Viton 377-9

Mechanical properties (contd)					Physical and thermal properties	
Rings (contd)		Compression set				
Tensile strength, psi	Elongation, %	t_0	t_1	Compression set, % $\left(\frac{t_0 - t_1}{t_0 - t_s} \times 100\right)$	Volume change, %	Weight change, %
2360	168					
2120	170					
2400	157					
(av) 2290	(av) 165					
2350	187				+1.620	+1.080
2380	185				+1.520	+1.073
1904	155				+1.843	+1.031
(av) 2200	(av) 176				(av) +1.661	(av) +1.061
1790	94	0.522	0.404	80.55	−0.613	+0.012
2120	111	0.525	0.406	79.18	+0.644	+0.034
2230	111	0.530	0.405	80.90	−0.177	+0.032
		0.524	0.405	80.13		
(av) 2050	(av) 105			(av) 80.19	(av) −0.395	(av) +0.026
1820	137	0.529	0.430	64.50	−0.530	+0.046
2430	156	0.5315	0.4285	66.03	+0.446	+0.030
1950	146	0.5335	0.4265	67.72	−0.210	+0.030
		0.5305	0.430	64.84		
(av) 2070	(av) 146			(av) 65.77	(av) −0.370	(av) +0.035
1910	122	0.510	0.411	73.6	+0.012	−0.069
2570	155	0.506	0.411	72.8	+0.047	−0.071
2280	138	0.504	0.411	72.4	−0.176	−0.102
		0.510	0.411	73.6		
(av) 2250	(av) 138			(av) 73.10	(av) −0.039	(av) −0.081
2790	217	0.532	0.424	69.0	−0.30	+0.100
2460	210	0.5305	0.4235	69.0	+2.11	+0.109
2200	200	0.525	0.421	69.5	+1.06	+0.123
		0.532	0.4285	66.1		
(av) 2480	(av) 209			(av) 68.4	(av) +0.92	(av) +0.111

Table B-3. Complete test results after ETO decontamination and dry-heat sterilization under various conditions for encapsulant Stycast 1090

Commercial designation	Cure schedule for unheated controls		Exposure conditions	Mechanical properties	Electrical properties			Physical and thermal properties	
	Duration, h	Temperature, °F		Hardness, Shore D	Thickness, mil	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
Stycast 1090/ catalyst 11	2	212	Control	72.9	102.7	1.66×10^{14}	424		
				70.5	104.3	1.25×10^{14}	431		
				71.9	102.8	1.22×10^{14}	447		
				(av) 71.8		(av) 1.38×10^{14}	(av) 434		
			ETO	71.5	101.3	5.6×10^{13}	405		+0.260
				70.1	103.0	7.8×10^{13}	304		+0.295
				70.1	103.4	8.2×10^{13}	397		+0.319
				(av) 70.6		(av) 7.2×10^{13}	(av) 381		(av) +0.291
			ETO + thermal (6 × 40 h at 155°C in N_2)	76.7	101.8	1.60×10^{14}	276	−0.368	−1.185
				76.7	103.9	2.03×10^{14}	289	−0.229	−1.173
				77.3	102.4	1.92×10^{14}	293	−0.361	−1.389
				(av) 76.9		(av) 1.85×10^{14}	(av) 286	(av) −0.319	(av) −1.249
			ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	75.3	103.9	1.71×10^{14}	337		−0.692
				74.7	103.1	1.70×10^{14}	335	−0.169	−1.002
				75.3	103.7	1.87×10^{14}	309	−0.180	−0.664
				(av) 75.1		(av) 1.76×10^{14}	(av) 327	(av) −0.175	(av) −0.786
			ETO + thermal (6 × 92 h at 135°C in N_2)	78	103	2.0×10^{14}			−0.715
				77	104	2.6×10^{14}		−0.490	−0.639
				76	103	1.8×10^{14}		−0.652	−0.524
				(av) 77		(av) 2.1×10^{14}		(av) −0.571	(av) −0.626
			ETO + thermal (6 × 600 h at 105°C in N_2)	76	102	4.4×10^{13}	284	−0.308	−0.254
				76	103	1.13×10^{14}	408	−0.379	−0.269
				76	104	1.25×10^{14}	320		−0.267
				(av) 76		(av) 9.4×10^{13}	(av) 337	(av) −0.343	(av) −0.263

Table B-5. Complete test results after ETO decontamination and dry-heat sterilization under various conditions for plastic laminate EG 758

Commercial designation	Exposure conditions	Mechanical properties				Electrical properties			Physical and thermal properties	
		Hardness, Rockwell E	Stressed dimensions, in.	Tensile strength, psi	Elongation, % (in 2 in.)	Thickness, mil	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
EG 758, type GEE	Control	86.7	0.128/0.501	41200	4.50	125.4	2.8×10^{15}	> 383		
		87.4	0.127/0.504	39100	3.25	125.7	3.5×10^{15}	> 382		
		86.7	0.126/0.500	40600	2.30	124.7	3.9×10^{15}	> 385		
		(av) 86.9		(av) 40300	(av) 3.35		(av) 3.4×10^{15}	(av) > 383		
	ETO	86.6	0.127/0.499	46900	1.90	125.5	4.9×10^{15}	> 382	0	+0.043
		88.2	0.128/0.497	47500	2.85	123.0	3.5×10^{15}	> 390	0	+0.055
		88.4	0.127/0.500	47400	1.65	123.6	5.4×10^{15}	> 389	0	+0.062
		(av) 87.7		(av) 47300	(av) 2.13		(av) 4.6×10^{15}	(av) > 387	(av) 0	(av) +0.053
	ETO + thermal (6 × 40 h at 155°C in N_2)	86.7	0.131/0.500	45500	2.20	118.6	1.32×10^{16}	> 405	−0.096	−0.323
		86.3	0.131/0.501	47100	2.30	121.2	1.40×10^{16}	> 396	+0.209	−0.245
		85.3	0.131/0.500	47900	1.80	129.3	1.13×10^{16}	> 371	−0.094	−0.201
		(av) 86.1		(av) 46800	(av) 2.10		(av) 1.28×10^{16}	(av) > 391	(av) 0.006	(av) −0.256
	ETO + thermal (6 × 92 h at 135°C in N_2 + 0.45% O_2)	89	0.131/0.502	47900	1.75	129.3	9.5×10^{15}	> 371	−0.096	−0.186
		88.8	0.131/0.500	46600	1.60	128.3	5.9×10^{15}	> 374	−0.122	−0.169
		89.7	0.130/0.499	46200	1.70	127.2	6.3×10^{15}	> 377	−0.219	−0.179
		(av) 89.2		(av) 46900	(av) 1.68		(av) 7.2×10^{15}	(av) > 374	(av) −0.146	(av) −0.178
	ETO + thermal (6 × 92 h at 135°C in N_2)	88.7	0.125/0.499	41700	1.45	125	1.56×10^{16}	384	+0.090	−0.191
		90.7	0.124/0.495	39900	1.45	133	1.20×10^{16}	361	−0.050	−0.181
		91.7	0.125/0.500	37800	1.40	125	0.98×10^{16}	384	+0.085	−0.234
		(av) 90.4		(av) 39800	(av) 1.43		(av) 1.25×10^{16}	(av) 376	(av) +0.042	(av) −0.202
	ETO + thermal (6 × 600 h at 105°C in N_2)	90	0.129/0.497	48200	1.45	128	4.1×10^{15}	375	−0.084	−0.097
		90	0.129/0.502	49400	1.45	129	4.1×10^{15}	372	−0.129	−0.089
		89	0.128/0.498	50500	1.40	126	3.6×10^{15}	381	−0.102	−0.121
		(av) 90		(av) 49400	(av) 1.43		(av) 3.9×10^{15}	(av) 376	(av) −0.105	(av) −0.102

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Table B-6. Complete test results after ETO decontamination and dry-heat sterilization at various conditions for tape Mystik 7352

Commercial designation	Exposure conditions	Mechanical properties				Electrical properties			Physical and thermal properties	
		Peel adhesion, oz/in. (5-in. av length separation)	Stressed dimensions, in.	Breaking strength, lb/in.	Elongation, % (in 3 in.)	Thickness, mil	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
Mystik 7352	Control	42.8	3	49	72	3.3	2.01×10^{15}	1960		
		45.0	3	48	67	3.3	1.53×10^{15}	1960		
		44.6	3	50	70	3.3	1.95×10^{15}	2020		
		(av) 44.1		(av) 49	(av) 70		(av) 1.83×10^{15}	(av) 1980		
	ETO	41.9	3	44	48	3.5	1.09×10^{15}	1855	0	+0.757
		44.2	3	45	53	3.5	1.50×10^{15}	1830	0	+0.727
		44.8	3	45	55	3.5	1.38×10^{15}	1820	0	+0.831
		(av) 43.6		(av) 45	(av) 52		(av) 1.32×10^{15}	(av) 1835	(av) 0	(av) +0.772
	ETO + thermal	88.6	3	44	57	3.5	9.2×10^{15}	1740	−1.852	−2.111
	(6 × 40 h at 155°C in N_2)	89.6	3	43	55	3.5	1.75×10^{16}	1770	−1.163	−2.105
		93.1	3	43	50	3.5	1.38×10^{16}	1840	−2.401	
		(av) 90.4		(av) 43	(av) 54		(av) 1.35×10^{16}	(av) 1780	(av) −1.962	(av) −2.108
	ETO + thermal	98.6	3	42	58	3.4	5.4×10^{15}	1900	−1.092	−1.723
	(6 × 92 h at 135°C in	97.3	3	40	52	3.4	6.6×10^{15}	1920	−0.681	−1.635
	N_2 + 0.45% O_2)	101.8	3	42	54	3.4	1.32×10^{16}	1840	−0.958	−1.177
		(av) 99.2		(av) 41	(av) 55		(av) 8.4×10^{15}	(av) 1887	(av) −0.910	(av) −1.511
	ETO + thermal	5.3	3			3.7	2.7×10^{16}	1830	−1.829	−1.190
	(6 × 92 h at 135°C in N_2)	5.6	3	40	56	3.6	3.0×10^{16}	1910	−2.660	−1.387
		4.8	3	41	57	3.6	1.5×10^{16}	1880	−2.277	−1.006
		(av) 5.2		(av) 41	(av) 56		(av) 2.4×10^{16}	(av) 1870	(av) −2.256	(av) −1.194
	ETO + thermal	80.0	3	46	83	3.5	7.8×10^{15}	1760	−0.316	−1.622
	(6 × 600 h at 105°C in N_2)	80.4	3	45	57	3.4	9.0×10^{15}	1850	−0.422	−1.692
		77.4	3	46	57	3.5	6.9×10^{15}	1780	−0.567	−1.473
		(av) 79.2		(av) 46	(av) 66		(av) 7.9×10^{15}	(av) 1800	(av) −0.435	(av) −1.595

Commercial designation	Exposure conditions
Tedlar, type 30, modification B, high gloss, 1 mil	Control
	ETO
	ETO + thermal (6 × 40 h at 155°C i
	ETO + thermal (6 × 92 h at 135°C i N_2 + 0.45% O_2)
	ETO + thermal (6 × 92 h at 135°C i
	ETO + thermal (6 × 600 h at 105°C

FOLDOUT FRAME 1

FOLDOUT FRAME #2

Table B-4. Complete test results after ETO decontamination and dry-heat sterilization under various conditions for film Tedlar 30

Mechanical properties					Electrical properties			Physical and thermal properties	
Tensile strength and elongation			Tear strength						
Stressed dimensions, in.	Tensile strength, psi	Elongation, % (in 1.0 in.)	Thickness, mils	Tear strength, lb/mil	Thickness, mil	Volume resistivity, Ω-cm	Dielectric strength, V/mil	Volume change, %	Weight change, %
0.38/0.0010	12200	123	1.0	1.95	0.9	2.0×10^{15}	3180		
0.38/0.0011	11700	146	1.0	1.92	0.9	1.2×10^{15}	3310		
0.38/0.0010	11400	132	1.0	1.62	0.9	1.7×10^{15}	3350		
	(av) 11800	(av) 134		(av) 1.83		(av) 1.6×10^{15}	(av) 3280		
0.39/0.0010	10300	107	1.0	0.98	0.9	1.5×10^{15}	3590	−0.889	+0.505
0.38/0.0010	11000	101	1.0	1.27	0.9	3.0×10^{15}	3460	−0.519	+0.525
0.38/0.0010	10200	166	1.0	1.56	0.9	3.1×10^{15}	3480	−1.039	+0.393
	(av) 10500	(av) 125		(av) 1.27		(av) 2.5×10^{15}	(av) 3510	(av) −0.816	(av) +0.474
0.40/0.0010	9930	87	1.0	1.60	1.0	1.5×10^{15}	3350	−11.459	−0.167
0.40/0.0010	9900	102	1.0	1.15	0.95	1.6×10^{15}	3640	−7.578	−0.126
0.45/0.0010	8730	104	1.0	1.10	0.95	1.2×10^{15}	3760	−8.488	−0.169
	(av) 9520	(av) 98		(av) 1.28		(av) 1.4×10^{15}	(av) 3580	(av) −9.175	(av) 0.154
0.40/0.0010	9800	80	1.0	1.58	0.9	1.26×10^{15}	3620	−5.188	+0.043
0.40/0.0010	10600	116	1.0	1.43	0.9	1.31×10^{15}	3700	−4.933	0
0.40/0.0010	10000	81	1.2	1.13				−4.916	0
	(av) 10100	(av) 92		(av) 1.38		(av) 1.29×10^{15}	(av) 3660	(av) −5.012	(av) +0.014
0.42/0.0008	9640	80	1.0	1.19	0.98	3.0×10^{15}	3400	−4.278	+0.041
					0.90	2.8×10^{15}	3710	−4.512	0
0.40/0.0008	12800	89	1.0	1.33	0.90	2.4×10^{15}	3690	−4.763	+0.086
	(av) 11220	(av) 85		(av) 1.26		(av) 2.7×10^{15}	(av) 3600	(av) −4.518	(av) +0.042
.0010/0.60	10400	118	1.0	1.12	0.90	7.5×10^{14}	3480	−2.799	−0.165
.0010/0.60	11500	135			0.90	1.78×10^{15}	3580	−3.956	−0.081
.0010/0.60	10900	116	1.0	1.33	0.98	—	3300	−3.008	−1.990
	(av) 10900	(av) 123		(av) 1.23		(av) 1.27×10^{15}	(av) 3450	(av) −3.254	(av) −0.740